925,380

PATENT SPECIFICATION

NO DRAWINGS

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COMPLETE SPECIFICATION

Improvements in the Production of Galactose

We, THOMAS KERFOOT AND CO. LIMITED, a British Company of Vale of Bardsley, Ashton-under-Lyne, County of Lancaster, do hereby declare the invention, for which we pray that a patent may be granted to us, and the method by which it is to be performed, to be particularly described in and by the following statement:—

This invention relates to an improved process for the production by hydrolysis of anhydrous D (+) galactose.

Anhydrous D (+) galactose is a hexose having the formula $C_6H_{12}O_6$ and a molecular weight of 180.1. It is a constituent of oligo saccharides such as lactose, melibiose, and raffinose and is obtained by the degradation thereof and a good yield of the substance can be obtained by the action of sulphuric acid on an aqueous solution of lactose at a temperature of 100°C.

Anhydrous D (+) galactose is used as a bio-chemical intermediate and as a stabiliser for injectable medicinal solutions, more particularly of iron (ferric) and related substances, and the main object of the present invention is the production from the crude galactose obtained by the degradation of the aforesaid oligo-saccharides of a substance having the purity required for use as a stabiliser for injectable solutions. A further object of the invention is a complete process for the production of crude galactose from lactose and the purification thereof.

The main object of the invention, i.e. the purification of crude galactose obtained by the degradation of an oligo-saccharide is achieved according to the present invention by dissolving the crude galactose in a methyl alcohol or ethyl alcohol or a mixture of water therewith, removing undissolved impurities from the resultant solution and recovering the purified galactose therefrom by crystallization.

The purified galactose may be recovered by direct crystallization from the solution or by evaporating the solution dissolving the purified material i.e. methyl alcohol or ethyl alcohol and then crystallizing out the galactose from the alcohol.

Alternatively, the crude galactose may be extracted with methyl alcohol or ethyl alcohol to form a solution of galactose therein, the solids separated from the solution, the resultant solution concentrated, the galactose allowed to crystallize, the crystals dissolved in methyl alcohol or ethyl alcohol and recrystallized therefrom.

The crude galactose may be produced by the hydrolisation of an aqueous solution of lactose with sulphuric or other mineral acid.

In the following examples, which are given to illustrate the carrying out of the invention, the complete process for the production of crude galactose from lactose and its subsequent purification is given but it is to be understood that the purification process can be applied to the purification of crude galactose obtained by the degradation of oligo saccharides by other processes, and further that the invention is not limited to the processes described in the examples.

EXAMPLE 1.

Tanks are suitably prepared free of all metal contamination and 227 volumes of a boiling 44% aqueous solution of lactose maintained and 44 volumes of a 10% V/V aqueous solution of sulphuric acid are added. The temperature is maintained at boiling point and allowed to hydrolyse for 2½ hours, or until a neutralized sample shows a specific rotation of $+\frac{20}{D}+67$—68. The hydrolystate is then neutralized with a suitable alkali, such as calcium carbonate or barium carbonate, (which precipitates to give the sulphate filtration). The solids are then allowed to settle.

The solution is clarified by passing through activated carbon or decolourising mats or by

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mixing decolourising carbon with the mass and filtering, and washed with water by decantation or any other similar method. The filtrate and the washings are concentrated under reduced pressure (1.54 at 20°C.), to a syrup, the syrup is then mixed by volume with glacial acetic acid, and allowed to cool to crystallization.

The liquors are then seeded with galactose crystals in order to start crystallization. After the first crystallization the mother liquors are separated by filtration or centrifuging, and the resultant liquors washed with 75% V/V industrial methylated spirits 64 op until free from traces of acetic acid. The final crystalline galactose is then oven dried at 65°C. The crystals are now in their crude state and re-crystallization is obtained as follows:

The crude galactose crystals are dissolved in 800 c.c. of distilled water and approximately 2,250 c.c. of industrial methylated spirits 64 op added. This crude solution is then refluxed for 1 hour. The final solution is filtered through decolourising carbon, allowed to cool and seeded with crystals of anhydrous D (+) galactose, the final product having the following characteristics and structural formula:

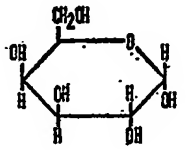
Optical Rotation:
of a 10% solution containing 0.2 mls. Ammonia Solution per 100 mls. is not less than +80° and not more than +81° (calculated on the dried material).

Loss on drying:
Not more than 0.5% in vacuo at 100°C.

Ash (sulphate):
Not more than 0.1%

Reaction:
A 10% aqueous solution is neutral to litmus.

Structure:



EXAMPLE 2.

100 volumes of a boiling 50% aqueous solution of lactose and 20 volumes of a 10% V/V aqueous solution of sulphuric acid are mixed and the solution allowed to react at 90°C., and hydrolise for 1 hour. The hydrolysate is then neutralized with a suitable alkali, as in Example 1, and the solids extracted immediately. Filtration, clarification and crystallization are then carried out as in Example 1.

Although 64 op industrial methylated spirits is used in the foregoing examples by reason of its cheapness, the invention can be carried out equally well by using other forms of methyl alcohol or by using ethyl alcohol. Moreover, the production of the crude galactose may be obtained from other oligo-saccharides, e.g. melibroise and raffinose by similar processes.

WHAT WE CLAIM IS:—

1. A process for the purification of crude anhydrous D (+) galactose produced by the degradation of an oligo-saccharide consisting in dissolving the crude galactose in methyl alcohol or ethyl alcohol or a mixture of water therewith, removing undissolved impurities from the resultant solution and recovering the purified galactose therefrom by crystallization.

2. A modification of the process claimed in claim 1 for the purification of crude anhydrous D (+) galactose produced by the degradation of an oligo-saccharide wherein the purified galactose is recovered by evaporating the solution, dissolving the residue in methyl alcohol or ethyl alcohol and then crystallizing out the purified galactose therefrom.

3. A process for the purification of crude anhydrous D (+) galactose produced by the degradation of an oligo-saccharide as in Claim 2 wherein the crystals produced are again dissolved in methyl alcohol or ethyl alcohol and re-crystallized therefrom.

4. A process for the purification of crude anhydrous D (+) galactose produced by the degradation of an oligo-saccharide as in any one of the preceding claims wherein the crude galactose is produced by the degradation of lactose with sulphuric or other mineral acid.

5. A process for the purification of crude anhydrous D (+) galactose produced by the degradation of an oligo-saccharide as in any one of the preceding claims wherein the alcohol employed is industrial methylated spirit.

6. A process for the purification of crude anhydrous D (+) galactose produced by the degradation of an oligo-saccharide as in Claim 3 wherein the crude crystals are dissolved in distilled water and methyl alcohol or ethyl alcohol added to the solution which is then refluxed, filtered through decolouring carbon and the purified galactose crystallized out.

7. A process for the purification of crude anhydrous D (+) galactose produced by the degradation of an oligo-saccharide substantially as hereinbefore described.

8. A process for the production of purified anhydrous D (+) galactose consisting in degrading an oligo-saccharide, e.g. lactose, melibiose or raffinose, with sulphuric or other mineral acid, neutralising the solution with a suitable alkali to cause precipitation to give a sulphate filtration, allowing the precipitate to

settle, clarifying the solution, concentrating it to a syrup, mixing the latter with glacial acetic acid, crystallizing out, separating the crystals, washing them with methyl alcohol or ethyl alcohol and finally re-crystallizing them by the processes claimed in any one of Claims 1 to 7.

9. A process for the production of purified anhydrous D (+) galactose substantially as described in Example 1 or Example 2 of the foregoing specification.

10. Anhydrous D (+) galactose when purified by the process claimed in any one of Claims 1 to 7.

11. Purified anhydrous D (+) galactose when produced by the process claimed in Claim 8 or Claim 9.

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